UNITED STATES

DEPARTMENT OF THE INTERIOR

BUREAU OF MINES

NASA Contract R-09-040-001

MULTIDISCIPLINARY RESEARCH LEADING TO UTILIZATION OF EXTRATERRESTRIAL RESOURCES

Annual Status Report
Fiscal Year 1970

TWIN CITIES MINING RESEARCH CENTER

Walter E. Lewis, Research Director

NASA Contract R-09-040-001

MULTIDISCIPLINARY RESEARCH LEADING TO UTILIZATION OF EXTRATERRESTRIAL RESOURCES

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U. S. Bureau of Mines NASA Program of Multidisciplinary Research
Leading to Utilization of Extraterrestrial Resources

ANNUAL STATUS REPORT FISCAL YEAR 1970

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U. S. Bureau of Mines NASA Program of Multidisciplinary Research Leading to Utilization of Extraterrestrial Resources

July 1, 1970

Task title:	Background analysis and coordination	
Investigator:	Thomas C. Atchison, Program Manager	
Location:	Twin Cities Mining Research Center Minneapolis, Minnesota	
Date begun:	April 1965	To be completed: Continuing
Personnel:	Thomas C. Atchison, Supervisory Research Physicist Other Bureau personnel, as assigned	

PROGRESS REPORT

Objective

The objective of the program is to help provide basic scientific and engineering knowledge needed to use extraterrestrial mineral resources in support of future space missions. Under this component, background and supporting studies and coordinating and liaison activities for the program are carried out.

Summary

During the year the six research tasks at the four research centers making up the Bureau's extraterrestrial resource utilization program were monitored. We continued to obtain, evaluate, and distribute information applicable to the program by literature search and direct contact with groups conducting related research. We also continued to extract and organize material from Bureau reports to provide periodic status reports to NASA and prepared special information related to the program when requested by NASA.

An informal review of the Bureau's program was held at Minneapolis in January. Continuation of the program into the sixth and seventh contract years was approved by NASA although funding was reduced from the rate of $300,000 per year to $250,000 per year because of overall reductions in NASA's budget.

Progress During the Year

Assistance to the experimental work of the program was continued by providing technical information and guidance to the task investigators. Following completion of all of the original research tasks last fiscal year, this year's effort was concentrated in six tasks, with primary emphasis on the more basic studies of material properties and behavior in simulated lunar environment. All of the tasks were planned for 2-year duration, but the study of electrowinning of oxygen from silicate rocks at Reno was terminated at the end of the year because of the reduction in funds.

Efforts continued on providing background information to task investigators with emphasis on results of the Apollo lunar surface experiments and returned lunar sample studies. T. C. Atchison and D. E. Fogelson took part in planning activities of the Working Group on Extraterrestrial Resources with meetings at NASA's Washington Headquarters, the Manned Spacecraft Center, Houston, Tex., the Air Force Institute of Technology, Dayton, Ohio, as well as at our Center in Minneapolis. The WGER is cooperating with Apollo program scientists to arrange a symposium scheduled for next fiscal year at the request of high NASA officials who are showing a growing interest in possible extraterrestrial resource utilization.

The usual formal review to evaluate progress on the Bureau's program was not held this year because of NASA travel fund limitations. Advantage was taken of the presence of several officers of the WGER, at our Center for a planning meeting, to conduct an informal review of the Twin Cities research tasks.

A proposal was prepared and submitted to NASA for continuing the Bureau's program into its sixth and seventh years. The proposal was accepted and funds have been made available for continuing the work at the level of $250,000 during fiscal year 1971.

Status of Manuscripts

Proposal for Continuing Bureau Extraterrestrial Resource Utilization Program by T. C. Atchison and NASA project leaders was submitted to NASA in December.

Strengths of Sulfur-Basalt Concretes by L. J. Crow and R. C. Bates was published as Bureau of Mines Report of Investigations 7349 in March.

Carbothermal Reduction of Liquid Siliceous Minerals in Vacuum by S. E. Khalafalla and L. A. Haas has been published in the Journal of High Temperature Science, v. 2, No. 2, June 1970, pp. 95-109.

Mineral Decomposition in High Vacuum by R. L. Carpenter was prepared for presentation at the Pacific Coast Regional Meeting of the American Ceramic Society at Seattle, Wash., October 15-17, 1969, and is being expanded for publication as a Bureau of Mines Report of Investigations.

Factors Related to Mineral Separation in a Vacuum by F. Fraas was published as Bureau of Mines Report of Investigations 7404 in June.

ANNUAL STATUS REPORT FISCAL YEAR 1970

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Surface properties of rock in simulated lunar environment
Investigator: Wallace W. Roepke, Project Leader
Location: Twin Cities Mining Research Center
Minneapolis, Minnesota
Date begun: April 1966 To be completed: June 1971

Personnel: William H. Engelmann, Supervisory Research Chemist
Wallace W. Roepke, Principal Vacuum Specialist
Kelly C. Strebig, Mining Engineer
Robert L. Schmidt, Mining Engineer
James R. Blair, Physical Science Technician
Bradley V. Johnson, Physical Science Aid

PROGRESS REPORT

Objective

The objective of this task is to study the surface properties of rocks and minerals in a simulated lunar environment. The study includes friction of metal-mineral pairs, drilling in rock, and shear testing of particulate mineral and rock materials.

Summary

This past year's effort emphasized mechanical development of the ultra-high vacuum (UHV) drill rig and development of proper test and operating techniques to provide valid test data. UHV drilling has shown effects which must be studied more closely. Some preliminary work was done in normal atmosphere on measuring drill-bit temperatures using a radiometer technique. The friction apparatus was substantially modified to provide more test capacity per pumpdown. A new UHV system for the friction work was received and placed in operation. Specifications were written on a consulting basis for Thermal Fragmentation for a small bench-top UHV system to be used for thermal properties studies. Specifications were also written for a data acquisition system to be used with the friction apparatus as an interface with the Center's new IBM 1130. Continuing gold vapor coating support work was provided for the scanning electron microscope.

Several trips were made in support of project work during the past year. On October 28-31, W. W. Roepke attended the 16th National Symposium of the American Vacuum Society in Seattle, Wash. During the same period, J. R. Blair attended a short course on vacuum techniques in Seattle, co-sponsored by the ISA and AVS. W. W. Roepke attended a Laser Power and Energy Measurement Course at the National Bureau of Standards, Boulder, Colo., December 4-5. On April 22-24, W. W. Roepke attended the 6th Annual Symposium of the New Mexico Section of the AVS in Albuquerque, N. Mex. The program at this symposium was devoted to surfaces and surface energy states. J. R. Blair completed a 100-hour night course in basic semiconductors at the St. Paul Area Technical Vocational Institute.

Progress During the Year

Ultrahigh Vacuum Drilling

Figure 1 shows the UHV drilling rig mounted in the chamber and operating during atmospheric collaring of a hole prior to regular drilling. During collaring, a vacuum cleaner nozzle is held near the drill bit to keep the ejecta from dropping into the bottom portion of the UHV system or into sensitive bearing areas. A high-speed camera used during some of the drilling tests is mounted on the tripod just to the right of the UHV system.

Figure 2 shows a closer view of the drilling rig immediately after an atmospheric test. Ejected material may still be seen piled around the drill bit. The rock sample is clamped to a turntable which can be remotely positioned and locked from outside the vacuum chamber. This feature is very helpful since pumpdown of the material most compatible with the UHV (dacite) requires 4 weeks to approach baseline pressure in the low 10^{-11} torr (low 10^{-9} N/m^2) region. Stainless steel plates are added to the top of the 47-lb (209 N) drill carriage to bring the total dead weight load on the drill to 106 lb (472 N). The pinion gear mounted just above the drill in the drill carriage has a ball bearing spline nut inside. The shaft running up through this pinion gear and spline nut is a three-channel spline shaft driven from outside the UHV chamber through a miter gear and a magnetically coupled rotary feedthrough. The miter gear pair is out of sight just below the plate where the spline shaft is pinned on the bottom end. Resting on the rock just to the left of the drill bit may be seen the end of the penetration measuring beam. This strain-gaged cantilever beam provides an electrical signal output as the bending moment increases due to drill penetration.

A great deal of time has been required to make the drilling rig UHV-compatible. Nearly all materials in the system are 300 series stainless steel to reduce outgassing sources other than the rock to an absolute minimum. Initially all bearings were in a clean, dry, and unlubricated condition but repeated and time-consuming bearing failures necessitated the use of a dry lubricant tungsten diselenide on some of the more critical bearings. The addition of this lubricant has increased the background gas species, and hence pumpdown time, appreciably.

The latest and most successful UHV drilling test to date was a 3-cm hole starting with a baseline pressure of 4.2×10^{-11} torr (5.6×10^{-9} N/m^2). The highest pressure reached during this test was 6×10^{-8} torr (8×10^{-6} N/m^2), but what is most significant is the fact that drilling actually

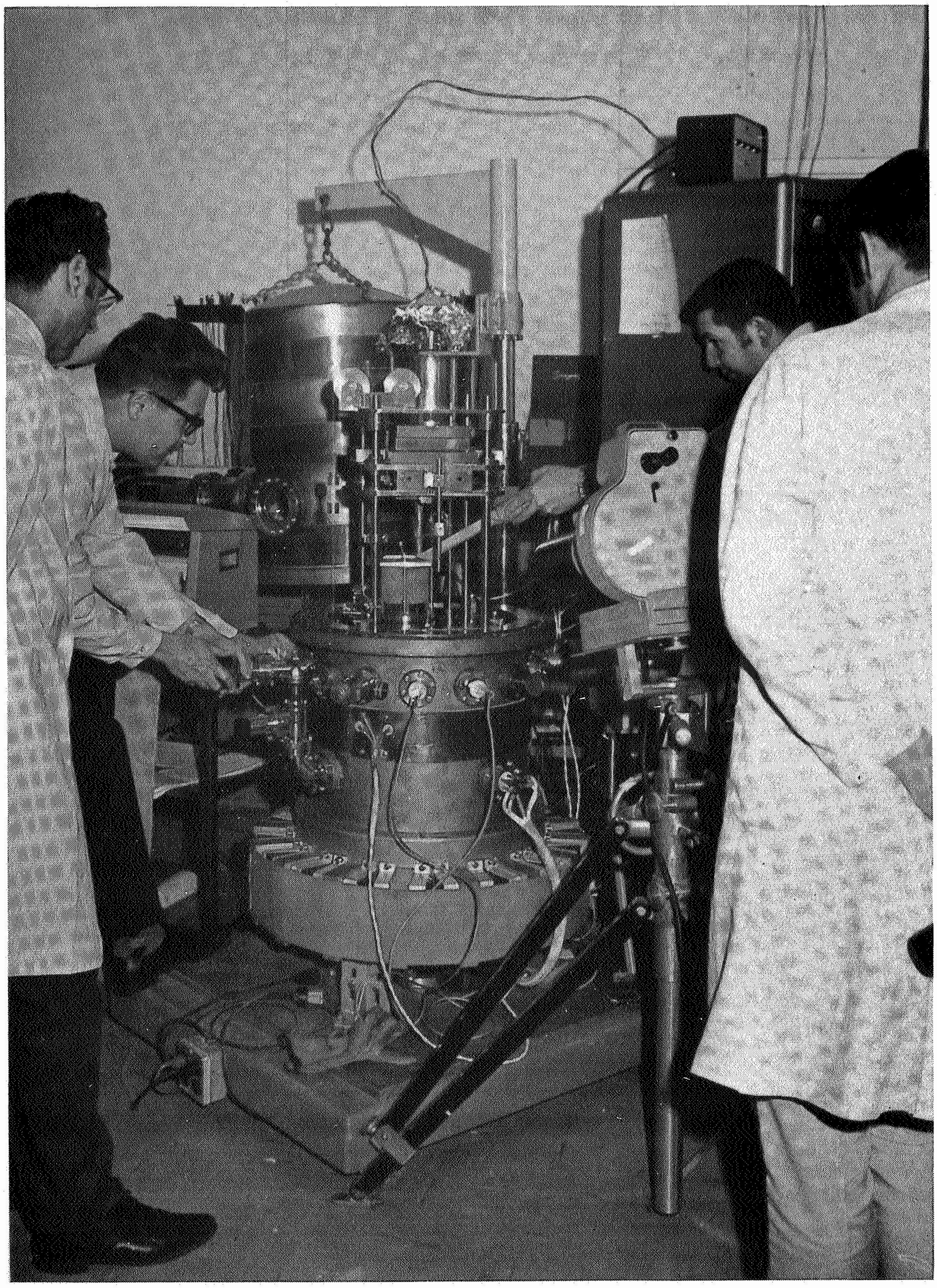

FIGURE 1. - Ultrahigh Vacuum Drilling Rig During Collaring Operation.

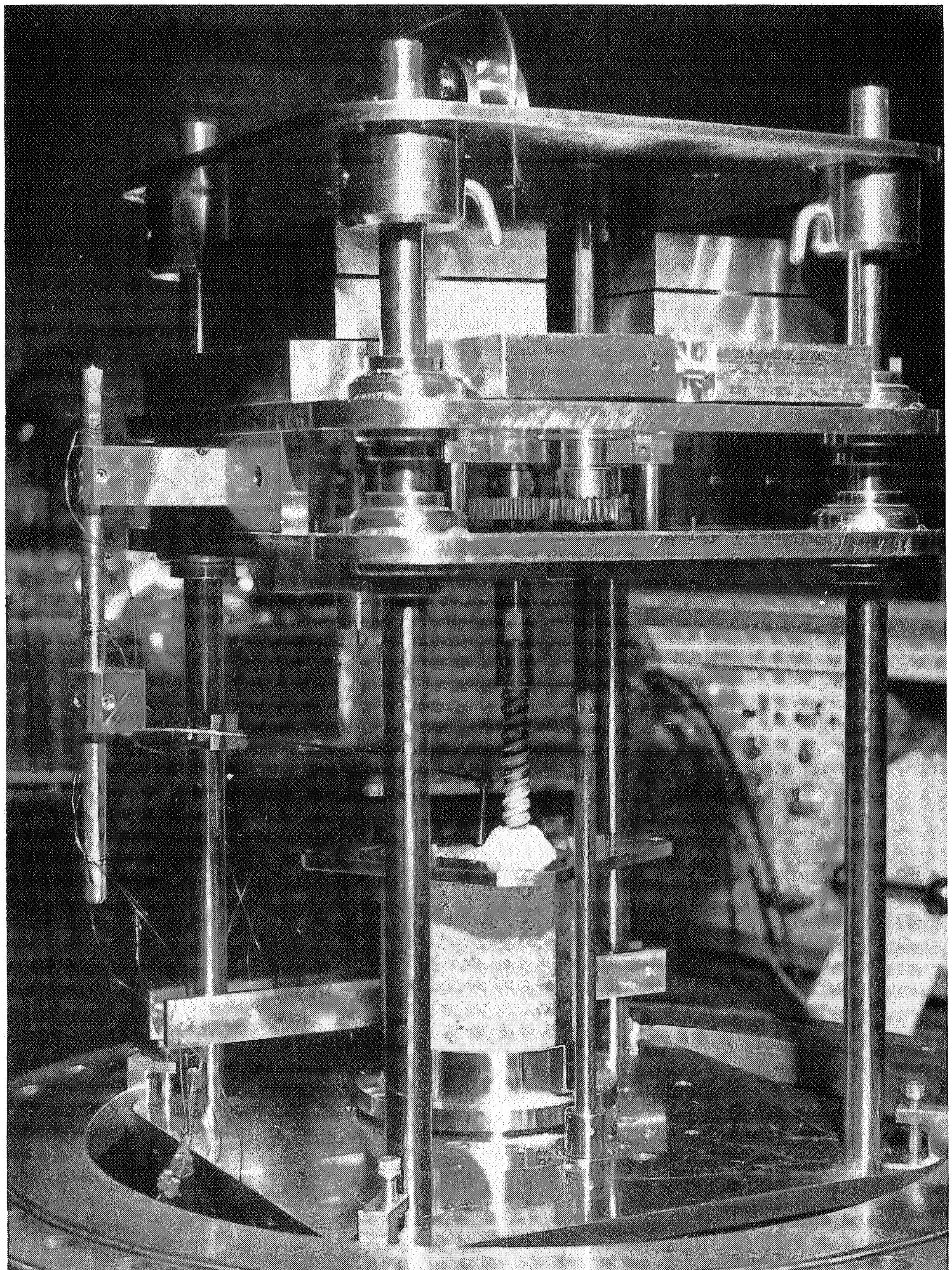

FIGURE 2. - Ultrahigh Vacuum Drilling Rig After Completion of an Atmospheric Drilling Test.

took place below 1.5 x 10^{-8} torr (2 x 10^{-6} N/m^2) for the major portion of the test. Unfortunately, failure of one of the main bearings precluded another test during this experiment. This latest test has shown, however, that a few minor corrections in technique will keep the system with a dacite sample in at least the 10^{-9} torr (10^{-7} N/m^2) range during drilling.

The results of this latest test indicate a 21 percent increase in energy input per unit volume (2π nt, where n = rpm and t = torque) for UHV drilling with a torque increase of 12.5 percent as compared to atmospheric drilling. Penetration during UHV drilling is 6 percent less than atmospheric and there is a 12 to 13 percent increase in the kinetic coefficient of friction for the UHV drilling.

These factors, becoming apparent in the first 1-1/4 inch (3.2 cm) of a laboratory test sample in UHV, do not indicate that drilling rock on the lunar surface is impossible. They do indicate, however, that great care may be required to prevent possible system damage from seizure, and further, that some artificial lubricant (gas ?) may be needed, down the borehole, to prevent particle agglomeration. Surface area analysis of cuttings from atmospheric and UHV drilling, using nitrogen gas adsorption techniques, shows the particle surface areas to be nearly the same in both conditions (0.874 m^2/g for particles produced in UHV and 0.902 m^2/g for particles produced in atmospheric conditions).

During atmospheric drilling to verify mechanical integrity of the drilling rig, some preliminary tests were made of rock and drill bit temperatures by remote (radiometer) sensing techniques. The test sample was positioned so that the drill bit traversed the outside edge of the sample breaking through the cylindrical surface. Temperature profiles were then recorded as the drill passed through the radiometer focal point. A temperature of 207° C maximum was observed at the drill-rock interface. As soon as the recorded curve indicated a decreasing temperature, drilling was stopped and the drill bit was returned to the radiometer focal point. The fastest return to this point after loss of contact with the rock was 3 seconds at which time a bit temperature of 291° C was recorded. The 12-mm focal spot of the radiometer caused an integration over some low temperature areas of drill and rock as well as the hottest portions of both. Additionally, all tests were run in an open room where convection would cause rapid temperature changes. The work looks very promising for estimating lunar drilling temperatures, but some refinements in technique will be required.

Ultrahigh Vacuum Friction Testing

The past year's effort on the friction work has been devoted to acquisition of equipment and redesign of the friction apparatus itself. Specifications were written and a new UHV system ordered in the first month of the fiscal year; however, this system was not received and accepted until the final month of the year. Although this delay severely curtailed the test program, it presented a good opportunity to redesign the friction test apparatus to increase its capability. The original design of the system is shown in figure 3, while figure 4 shows the modified version. In figure 4 the wheel is positioned higher which places the test sample in line with the germanium window for laser cleaning of the surface. The major redesign is in the probe carrier mechanism (figure 5). In the previous version we were limited to horizontal movement allowing the probe to be loaded or unloaded in a single track on each sample. This meant that we had only 11 test opportunities for each pumpdown since the sample wheel holds 11 samples. The redesign added vertical motion capability to the previously available horizontal drive providing more stability and increasing the number of tests per pumpdown from 11 to a minimum of 264. In figure 5 the bottom fingers are holding the horizontal driving shaft. This shaft and the driven one above it (by chain and sprocket on right end) move the whole probe assembly in and out for loading or sample change. Total horizontal displacement is greater than 1 in. The vertical driving shaft is at bottom center. This assembly lifts or lowers only the probe mounting bracket through a total displacement of approximately 1 in. The additional capacity of the fixture has created a data handling problem which we expect to solve by using the recently acquired IBM 1130 computer. Specifications have already been written for a data acquisition system which will allow digital magnetic tape recovery of the analog data generated by the friction apparatus. Once the proper programming is in the computer memory we will shorten our data handling from days to minutes.

Ultrahigh Vacuum Vane Shear Testing

The UHV portion of the vane shear testing program is being performed at this Center as a cooperative effort with the Spokane Mining Research Laboratory. The UHV vane shear apparatus has been fabricated, assembled, and bench tested this fiscal year. Since the vane shear equipment uses the same support frame in the UHV system as the drilling rig, there has been an unavoidable scheduling problem. The vane shear testing has therefore been restricted to the bench testing of components. Figure 6 shows the vane shear apparatus fitted to the UHV drilling frame. The system has three major sections: support frame, vane shear head with torque sensor, and sample holder with load cell. A fourth component, the powder bed stirring apparatus, is not in the photograph but during use mounts on that portion of the vane shear head which is just above the sample holding chamber.

Status of Manuscripts

Mass Spectrometer Studies of Outgassing From Simulated Lunar Materials in Ultrahigh Vacuum by W. W. Roepke and C. W. Schultz is being revised for publication in the Journal of the American Vacuum Society.

Friction Tests in Simulated Lunar Vacuum by W. W. Roepke was submitted to NASA for publication in the Proceedings of the Seventh Annual Meeting of the Working Group on Extraterrestrial Resources.

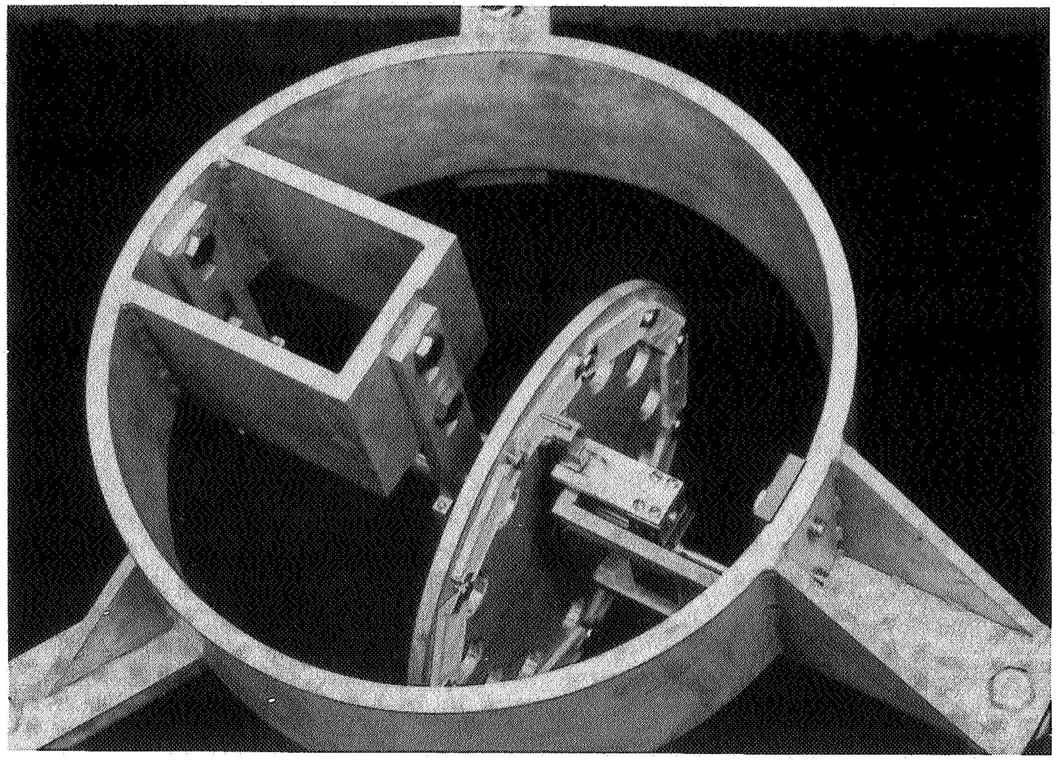

FIGURE 3. - Early Version of Ultrahigh Vacuum Friction Apparatus.

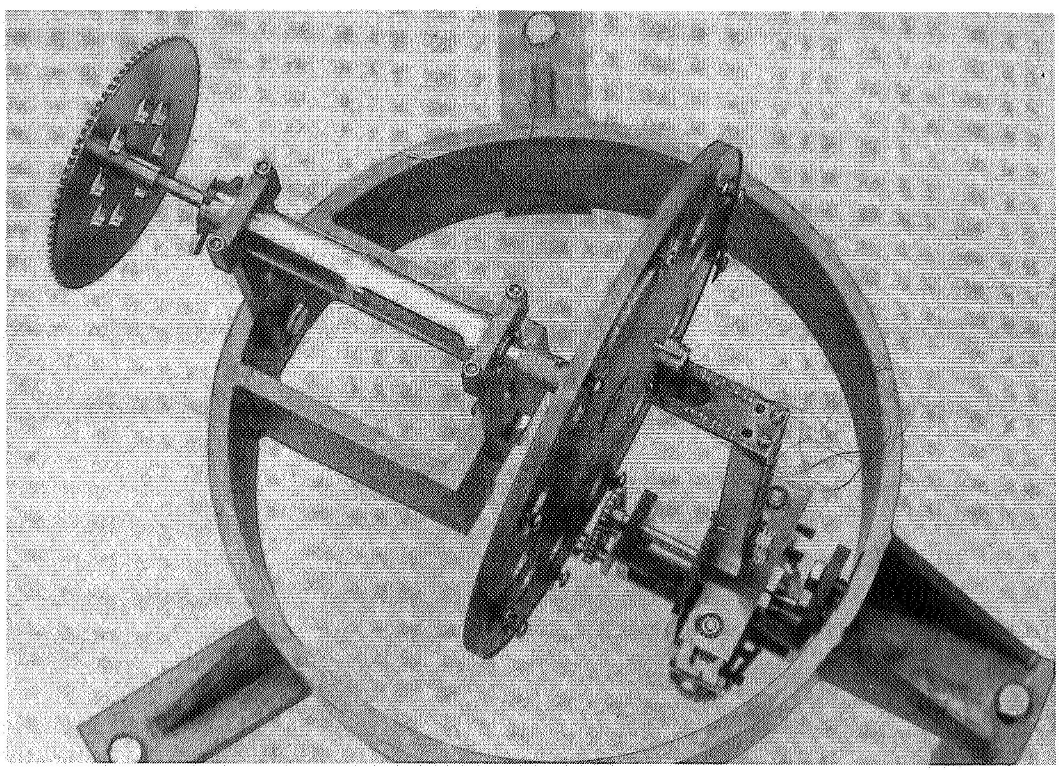

FIGURE 4. - Modified Version of Friction Apparatus Showing Arrangement for Laser Cleaning of Samples and Multitrack Probe Holder.

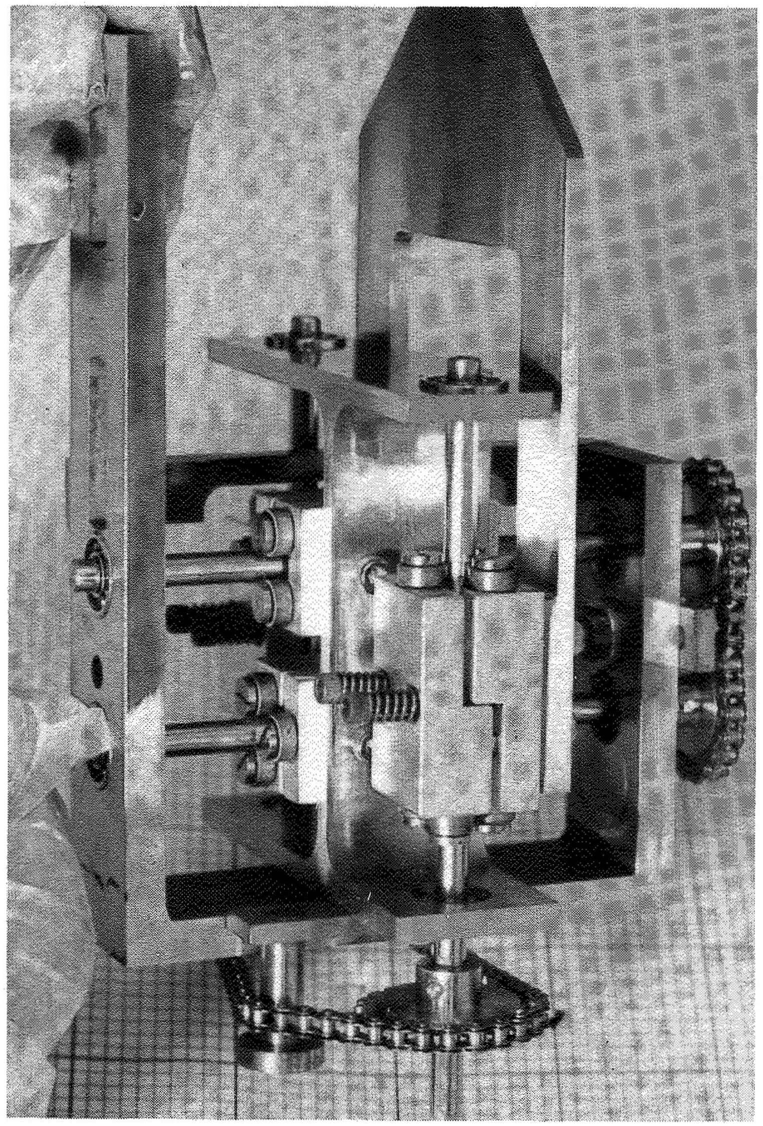

FIGURE 5. - Friction Probe Holder as Modified for Multitrack Testing.

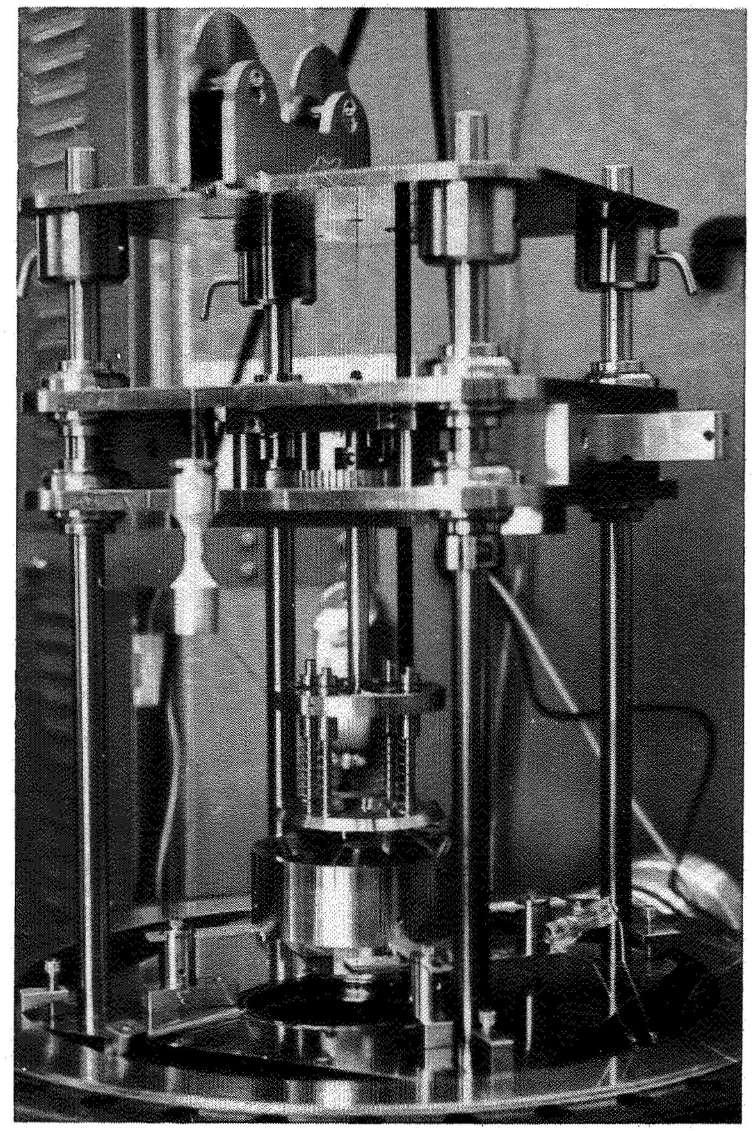

FIGURE 6. - Ultrahigh Vacuum Vane Shear Apparatus Fitted to Drilling Rig Frame for Compatability Testing.

Developing a Lunar Drill: A 1969 Status Report by R. L. Schmidt was submitted to NASA for publication in the Proceedings of the Seventh Annual Meeting of the Working Group on Extraterrestrial Resources.

IR-Visible Window Composite for UHV by W. W. Roepke was published in Review of Scientific Instruments, v. 41, No. 2, 1970, pp. 243-244.

Suppression of Mass Spectrometer Generated Interference on a Nude Bayard-Alpert Gauge in UHV by W. W. Roepke and K. G. Pung is being prepared as a journal article.

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Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Rock failure processes and strength and elastic properties in simulated lunar environment
Investigator:	Peter G. Chamberlain, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis, Minnesota
Date begun:	June 1966 To be completed: June 1971
Personnel:	Egons R. Podnieks, Supervisory Mechanical Engineer Robert J. Willard, Geologist Thomas R. Bur, Geophysicist Richard E. Thill, Geophysicist Peter G. Chamberlain, Geophysicist Laxman S. Sunda, Mining Engineer Kenneth G. Pung, Physical Science Technician Rollie C. Rosenquist, Engineering Technician

PROGRESS REPORT

Objective

The objective of this project is to study the effect of simulated lunar environment on rock deformation and failure processes at the macrostructural and the microstructural level. Rock strength and elastic properties of simulated lunar rocks will be determined in ultrahigh vacuum at temperatures over the lunar surface temperature range.

Summary

Compression tests were performed on three rocks from our suite of simulated lunar rocks in ultrahigh vacuum. Vacuum effects indicated a significant strength increase for tuff but not for dacite; the large variation in basalt data prevented meaningful statistical treatment of vacuum effects. Rock fabric was compared with results of last year's pulse velocity anisotropy tests on each of the simulated lunar rocks to determine fabric parameters controlling anisotropy. For most of the rocks, preferred orientation of elongate vesicles appeared to be the predominant factor.

Progress During the Year

Compression Tests in Ultrahigh Vacuum

Compression tests were performed on basalt, tuff, and dacite at room temperature in ultrahigh vacuum. Test results were compared with data from similar tests performed at atmospheric pressure (table 1) using statistical analysis (t-test) to determine the significance of vacuum effects on the measured properties.

TABLE I. - Compression test data for tuff, dacite, and basalt

Test condition	Number of tests	Compressive strength 10^3 psi	Compressive strength MN/m^2	Young's modulus 10^6 psi	Young's modulus GN/m^2
TUFF					
Room condition[1] ...	-	0.85	5.9	0.3	2
Directly in N_2	4	1.09	7.5	.34	2.36
N_2 after ultra-... high vacuum pumpdown	4	1.60	11.0	.39	2.70
Ultrahigh vacuum..	5	1.74	12.0	.38	2.62
DACITE					
Room condition[1] ...	-	6.0	41	2.0	14
Directly in N_2	4	6.19	42.7	1.97	13.6
N_2 after ultra-... high vacuum pumpdown	3	7.07	48.8	2.13	14.7
Ultrahigh vacuum..	5	7.09	48.9	2.35	16.2
BASALT					
Room condition[1] ...	-	53.0	365	10.3	70.9
Directly in N_2	4	48.1	332	19.5	134
Ultrahigh vacuum.. (2-day pumpdown)	5	42.8	295	17.9	123

[1]From "Simulated Lunar Rocks" by David E. Fogelson, Proceedings of the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources, NASA SP-177, 1968, pp. 75-95. Properties were run on different blocks than the ones used for the tests being reported here.

Tuff had a significantly higher compressive strength in ultrahigh vacuum than in a dry environment created by surrounding the core with dry nitrogen. Young's modulus also appeared higher in vacuum than in dry nitrogen but the difference between the modulus in the two environments was not statistically significant. The effect of vacuum on the compression properties is probably related to increased moisture removal caused by vacuum; specimen outgassing and chamber pressure data support this contention.

To check whether the strength increase was due to vacuum per se or was instead caused by increased water vapor removal by vacuum, several specimens were evacuated identically as those tested in ultrahigh vacuum and then backfilled with dry nitrogen prior to testing. Statistical analysis of the results of compression tests indicated these specimens were as strong as those tested in ultrahigh vacuum. This would indicate that the strength increase under vacuum conditions is brought about by removal of water vapor and other gases from the specimen which are not replaced if the specimen is backfilled with laboratory-grade dry nitrogen.

For dacite both the compressive strength and Young's modulus were slightly higher in ultrahigh vacuum than in nitrogen; however, the differences were not significant at the usual 90 percent confidence level (table 1). The lack of significant vacuum effect on dacite is due to the high permeability which allows most of the moisture to be removed during the standard preconditioning procedure (applied to all cores) which involves a medium vacuum ($\approx 50 \times 10^{-3}$ torr) evacuation. During compression tests only slight increases in chamber pressure were noted as the core fractured indicating that the core interior is well evacuated.

Basalt data (table 1) showed such a large variation that no significant trend can be established. The basalt is so impermeable that relatively little gas is removed during a 2-day pumpdown to ultrahigh vacuum. Pressure and mass spectrometer data support this conclusion; upon specimen fracture in the vacuum chamber, a heavy gas load was spontaneously released from the interior of the specimen. In hopes that a longer pumpdown will remove enough of the gases trapped within basalt specimens so that the effect on compression properties can be realistically determined, several specimens have been placed in the chamber for a long-term pumpdown.

Because of the long pumpdown being applied to the basalt specimens, compression testing of selected rocks in vacuum at the lunar temperature extremes has not progressed quite as far as originally expected. Recently increased facilities for performing compression tests should allow us to complete this phase of the experimental work along with the work scheduled for FY 71 by the June 71 deadline without difficulty. For the temperature tests, cores of dacite and basalt have been prepared, preconditioned and acoustically tested to determine nondestructively dynamic elastic moduli preparatory to compression testing in the vacuum chamber. Dummy specimens of each rock type have been instrumented with thermocouples for monitoring specimen temperature, and techniques for obtaining a given specimen temperature in the vacuum chamber are being refined.

A report, "Method for Rock Property Determination in Ultrahigh Vacuum," was drafted and sent out for review. A report on the effects of vacuum on compression properties of rock has been initiated. E. R. Podnieks attended a course in vacuum technology at UCLA February 16-27.

Anisotropy Studies

Pulse velocity and attenuation symmetry patterns determined for the suite of simulated lunar rocks last fiscal year were analyzed in more detail (table 2). The attenuation symmetry was the same as the elastic symmetry (determined from the velocity pattern) for all of the rocks except dunite and tuff. The difference between the elastic and attenuation symmetries of these two rocks indicates that the major controlling subfabric of one property possesses a symmetry system (for dunite) or an orientation (for tuff), differing from that of the major controlling subfabric of the other property.

TABLE 2. - Elastic and attenuation symmetries of simulated lunar rocks

Rock	Elasticity		Attenuation	
	System[1]	Anisotropy[2] (percent)	System[1]	Anisotropy[2] (percent)
Pumice	0	108	0 or TI	155
Vesicular basalt #3	0	43	0	155
Rhyolite	0	36	0	100
Serpentinite	0	27	0	185
Dacite	0	27	0	85
Semiwelded tuff	0	25	0	100
Dunite	TI	15	0	65
Altered rhyolite	0	13	0 or M	100
Granodiorite	0	10	0	65
Vesicular basalt #1	0	6.7	0	65
Duluth gabbro	0	3.3	0	35
Obsidian	TI	.7	([3])	([3])
Tholeiitic basalt	I	0	([3])	([3])
Vesicular basalt #2	H	-	-	-

[1]H = heterogeneous, I = isotropic, M = monoclinic, 0 = orthorhombic, TI = transversely isotropic.

[2]Calculated from $2(V_{max} - V_{min})/(V_{max} + V_{min})$ for elasticity and a similar equation for attenuation.

[3]Resolution was not good enough to obtain these values.

Determination of the anisotropic elastic constants will be carried over into next year so that the tests can be performed in vacuum. Due to the additional time required to perform tests in vacuum, representative rock types will be tested instead of the entire suite as originally proposed.

A report on elastic and attenuation symmetries of the simulated lunar rocks was drafted and sent out for review.

Fabric Analysis

Rock fabric is being compared with pulse velocity symmetry patterns previously obtained from spheres of the lunar rock. A thin section was cut from each sphere along the high-low velocity plane established from the pulse velocity measurements. Defect analysis of these thin sections showed that, for most rocks, elongate vesicle alignment seemed to be the chief fabric parameter controlling velocity symmetry. Alignment of elongate plagioclase grains in gabbro and oriented microcracks in fresh rhyolite seem to determine their symmetry.

The defect anlaysis was unable to establish the contribution of different subfabrics for dunite, granodiorite, and serpentinite; therefore, an additional analysis will be made with the universal stage. Dunite specimens have already been prepared for this purpose.

A series of photographs of thin sections from five of the lunar rocks were sent to Dr. H. Pincus at the University of Wisconsin-Milwaukee for optical data processing. Optical data processing provides another way of analyzing directional fabric features in these rocks for comparison with fabric analysis techniques employed at Twin Cities Mining Research Center and with results of velocity anisotropy studies.

Samples of flood basalt and dacite were coated with gold in preparation for a scanning electron microscope study aimed at comparing surface features that would explain their mechanical behavior during compression tests and their contrasting degassing behavior in vacuum.

The paper, "Effect of Moisture and Temperature on the Fracture Morphology of Dacite," was drafted and is through station review.

Status of Manuscripts

Environmental Effects on Rock Properties by E. R. Podnieks, P. G. Chamberlain, and R. E. Thill has been submitted for publication in the Proceedings of the Tenth Symposium on Rock Mechanics held at the University of Texas in May 1968.

Elastic and Attenuation Symmetries of Simulated Lunar Rocks by T. R. Bur and K. E. Hjelmstad is being prepared as a Bureau of Mines Report of Investigations or journal article.

Effect of Moisture and Temperature on the Fracture Morphology of Dacite by R. J. Willard and K. E. Hjelmstad is being prepared as a Bureau of Mines Report of Investigations or journal article.

Method for Rock Property Determination in Ultrahigh Vacuum by E. R. Podnieks and P. G. Chamberlain is being prepared for presentation and publication in the Proceedings of the ASTM/IES/AIAA Space Simulation Conference in September 1970.

ANNUAL STATUS REPORT FISCAL YEAR 1970

Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Thermal fragmentation and thermophysical and optical properties in simulated lunar environment
Investigator:	David P. Lindroth, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis, Minnesota
Date begun:	July 1969 To be completed: June 1971
Personnel:	Kuppusamy Thirumalai, Mining Engineer David P. Lindroth, Physicist Sam G. Demou, Physicist Walter G. Krawza, Engineering Technician

PROGRESS REPORT

Objective

The objective of this work is to study the problems of thermal fragmentation in lunar environment. Through the use of nondestructive testing and remote sensing technology, the thermophysical properties of simulated lunar rocks are to be determined as a function of temperature and pressure over the lunar environment range. Also, the optical properties of absorptance, reflectance, transmittance, and the absorption coefficient are to be determined as a function of wavelength and temperature.

Summary

Work during the year dealt with design and development of a system to be used for the determination of diffusivity, conductivity, and heat capacity by means of the flash method. A literature search was conducted on the optical properties of rock to determine the wavelengths best absorbed by the material and to determine the absorption at a wavelength of 10.6 μm. Experiments on thermal fragmentation in vacuum by CO_2 laser irradiation and on surface layer thermal expansion, begun last year under this task, were completed and the results were presented at the 1970 Symposium on Thermal Expansion of Solids at Santa Fe, N. Mex., in June.

Progress During the Year

The literature search to obtain data on the absorptance, absorption, coefficient, transmittance, and reflectance as a function of temperature and wavelength has been completed. This search was initiated to determine which type of currently available laser is optimal for the thermal fragmentation of rock. The majority of the data found covers the wavelength range from 0.4 to 30 μm. Data on the variation of absorptance as a function of temperature were scarce. The data obtained from the search show major absorption occurring around wavelengths of 10.8 and

23.0 μm. These strong absorption peaks are due to the characteristics of silicon and oxygen. Since the majority of the simulated lunar rocks, as well as other rock types under study, have a large percentage (50 percent or greater) of SiO_2, these wavelengths (10.8 and 23.0 μm) are of major importance as far as efficient energy input to the rock is concerned. After looking at the lasers currently available and the output wavelengths of each type, we conclude that the high power output of the CO_2 laser at a wavelength of 10.6 μm is optimal at this time for thermal rock fragmentation studies.

The design and fabrication of a multispecimen sample holder to be used for thermal property measurements by the flash method has been completed. The sample holder was made from No. 304 stainless steel in the form of a wheel to accommodate 10 specimens at one time. This sample holder will be used for the low temperature measurements. Currently underway is the design of a small tube furnace which will accommodate one specimen at a time for the high temperature measurements up to 1,000° K.

In order to obtain the thermal diffusivity by the flash method the sample thickness and the temperature rise versus time at the rear face of the sample are required. These parameters are relatively easy to obtain. However, to obtain the thermal diffusivity plus the thermal conductivity and heat capacity at the same time on the same sample requires that the energy density absorbed at the front face of the sample be known. This creates two problems. First, the absorption must be known as a function of temperature and wavelength for the sample under test. This problem has been partially solved by the findings from the literature search; however, much work remains to be done in this area. The second problem existing is that of accurately measuring the output energy from the source being used. Since we are using a pulsed CO_2 laser as the energy source this presents some difficulty.

The techniques necessary for measuring the output energy from a pulsed CO_2 laser were studied during the year. There is as yet no measurement standard for energy from a pulsed CO_2 laser. The CO_2 laser power and energy measurements are presently made with devices which convert radiant energy to heat and measure a change in temperature. These devices are termed thermopiles, detectors, balometers, and calorimeters. Calorimeters of the type that operate in a constant temperature environment can be analyzed in terms of a common heat flow problem. Thus the calorimeter technique offers advantages. However, since the problems of laser power and energy measurements are so new, some of the measurement ideas used in calorimetry have not yet been adopted as standard. These conclusions were confirmed in a seminar on Laser Power and Energy Measurements attended by the project leader at the National Bureau of Standards, Boulder, Colo.

For power measurements, we are using a power meter which is accurate to ± 5 percent for pulse durations longer than 1 second, which is adequate for the current work. The most recent power meter used by the National Bureau of Standards had an accuracy of approximately 3 percent at 100 watts. For the measurement of energy, NBS has build calorimeters to measure the output of ruby and neodymium lasers. However, there are no energy meters currently available for measuring the pulsed output of the CO_2 laser. We will have to have an energy meter custom built for this work.

High purity alumina (Al_2O_3) has been tentatively selected as a standard material for use with the flash method in determining the accuracy of the system. This material was chosen because of its high thermal shock resistance and also because of the large amount of thermal property data in the literature. Alpha-alumina (Al_2O_3) has also been used as a calorimetry standard by the National Bureau of Standards and has been used by us to determine the accuracy of our calorimeter.

Due to the high priority placed on drilling and other studies in ultrahigh vacuum, the apparatus for the flash method was not set up and no determination on the overall system precision and accuracy has yet been made. Because of the quantity of work scheduled for the UHV systems, it is not feasible to perform the thermal property and friction studies in the same UHV chamber as originally planned. We have therefore decided to purchase a small bench-top ultrahigh vacuum system to be used specifically for the thermal property studies. The specifications for the system have been drawn up and the paper work has been completed for the bidding process.

A study of rock response to heating was carried out during the year. Thermal expansion forms a prominent thermophysical property governing the generation of thermal stresses during heating of a rock material. A study of thermal expansion behavior and the response of rock material to induced thermal stresses is therefore necessary for an understanding of rock reaction to heating. Studies of the effect of reduced pressure on thermal fragmentation of rocks were also carried out during the year.

In order to enable a simple analysis, uniform incident heat flux on the rock surface was considered. The thermal gradients induced during heating and relative differences in thermal expansion and thermoelastic properties of the rock material generate internal thermal stresses. The anisotropism of the individual grains augments the generation of internal thermal stresses. Since the individual grains are constrained from thermal expansion, the internal stresses store thermoelastic energy. A release of the stored energy takes place in the form of inelastic response of the rock material. Such inelastic response will consist of immediately irrecoverable changes in the rock material, including the formation and/or extension of microcracks. Their magnitude at any temperature will therefore represent the extent of internal damage caused by heating and cooling, or in other words, the reaction of rock material to induced thermal stresses.

An ideal example of thermal expansion cycling for a constant rate of heating to temperature T_1 and cooling to the initial temperature is shown in figure 1. The total thermal dilation, ϵ_{total}, at any point C for

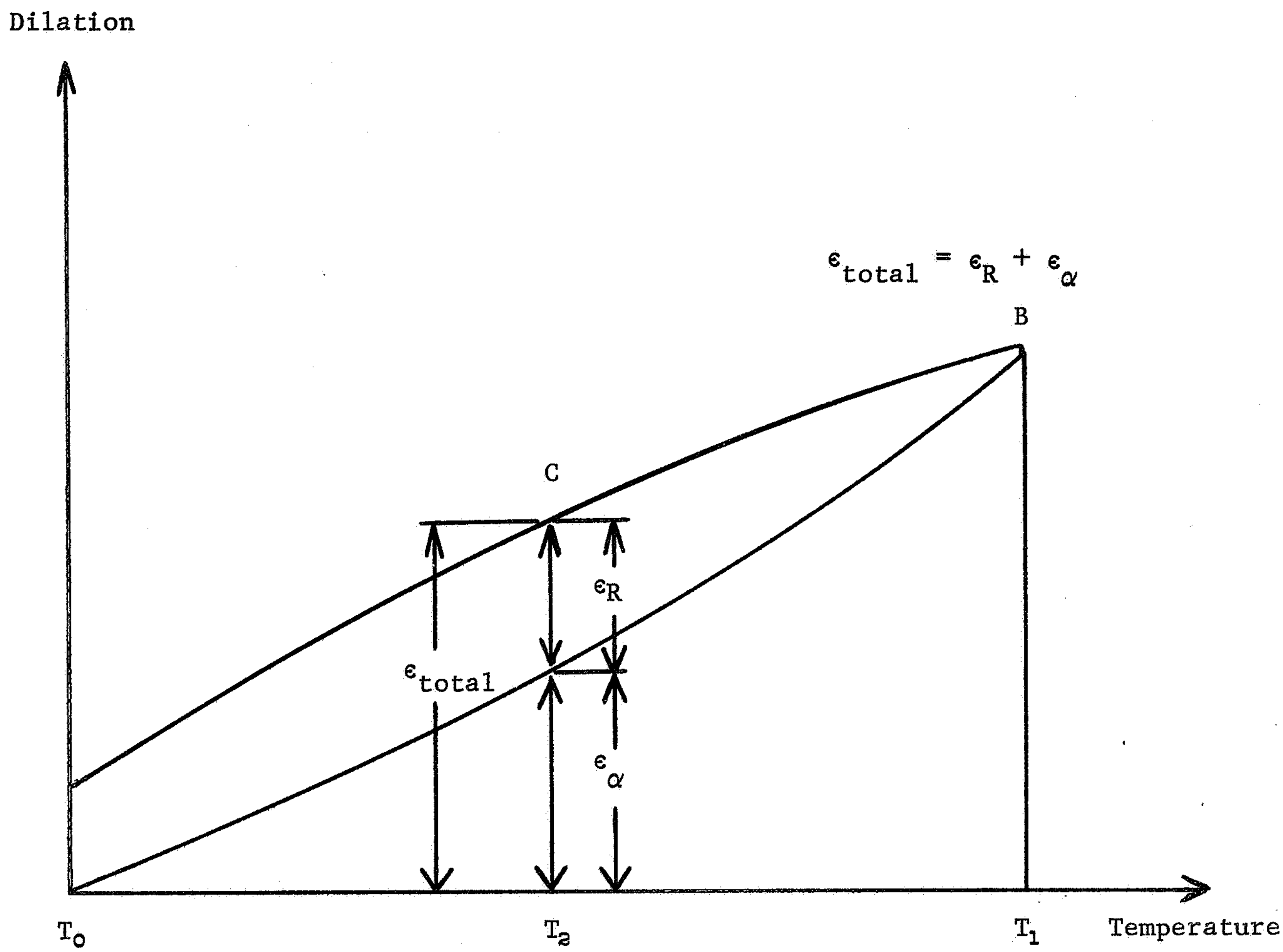


FIGURE 1. - An Ideal Thermal Expansion Cycling of a Rock Material.

temperature T_2, can be considered as the sum of observed expansion, ϵ_α, during heating and immediately irrecoverable thermal dilation, ϵ_R. The structural damage of the rock material induced by internal thermal stresses is proportional to the magnitude of ϵ_R. An experimental study was carried out to evaluate these parameters in rocks with different magnitudes of thermoelastic anisotropy. Because of NASA interest in the effect of reduced pressure on these parameters, two simulated lunar rock materials, granodiorite and obsidian, were used in the study along with Sioux Quartzite. Table 1 shows the crystallographic system of major mineral constituents, number of elastic constants, and the variations in thermoelastic properties of the major mineral constituents. An analysis was carried out to determine the relative order of susceptibility of these test rocks to generate internal thermal stresses during heating or cooling.

Since the surface of the rock specimen is exposed to maximum temperature differences during heating or cooling, coordinated temperature and thermal dilation measurements of the surface were necessary for the study. A new technique was evolved to obtain the experimental measurements using high temperature strain gages. The strain gage was bonded to the surface of 2- by 1/2- by 1/2-inch (5- by 1.3- by 1.3-cm) test specimens with a plasma spray of molten aluminum oxide. An identical strain gage along with a thermocouple was bonded to a similar specimen of fused quartz to compensate for environmental effects. The gages were connected in a wheatstone bridge. Due to the homogeneous, isotropic, and amorphous structure of fused quartz, the effect of internal thermal stresses is negligible. The output of the strain gage circuit is proportional to the difference in thermal dilation between the test rock and fused quartz. The standard values for fused quartz are added to obtain the data for the test rocks.

Figure 2 shows an example of thermal expansion measurements of the test rocks in a dry nitrogen atmosphere and reduced pressure down to 10^{-5} torr (10^{-3} N/m^2). The mean of six experimental readings along with standard deviations are presented in the figure. Among the test rocks, Sioux Quartzite shows the maximum percentage of thermal expansion, followed by granodiorite and obsidian, in dry nitrogen atmosphere and in reduced pressure. A statistical analysis of the experimental data shows no significant variations between the thermal expansion of test rocks in atmosphere and reduced pressure.

Figure 3 shows as an example the results of experimental analysis of the magnitudes of immediately irrecoverable dilation, ϵ_R, of granodiorite and obsidian. The higher magnitudes of ϵ_R in granodiorite than obsidian agree with the analysis based on the differences in thermoelastic properties of the major constituents of the rock material described in table 1. An analysis of the experimental data shows no significant differences between the readings obtained in the nitrogen atmosphere and in reduced pressure.

TABLE 1. - Description of test rocks

Test rocks	Major mineral constituents, percent of total volume	Crystal system	Thermoelastic properties			
			Number of elastic constants	Thermal expansion		
				Number of variations	Percent expansion at 200° C	
					Maximum	Minimum
Granodiorite	Plagioclase, 40	Monoclinic	13	4	0.22	0.06
	Quartz, 40	Hexagonal	5	2	.30	.18
Sioux Quartzite	Quartz, 98	Hexagonal	5	2	.30	.18
Obsidian	Glass, 65	Amorphous	1	none	.28	.28
	Microlites, 30	Isometric	3	none	--	--

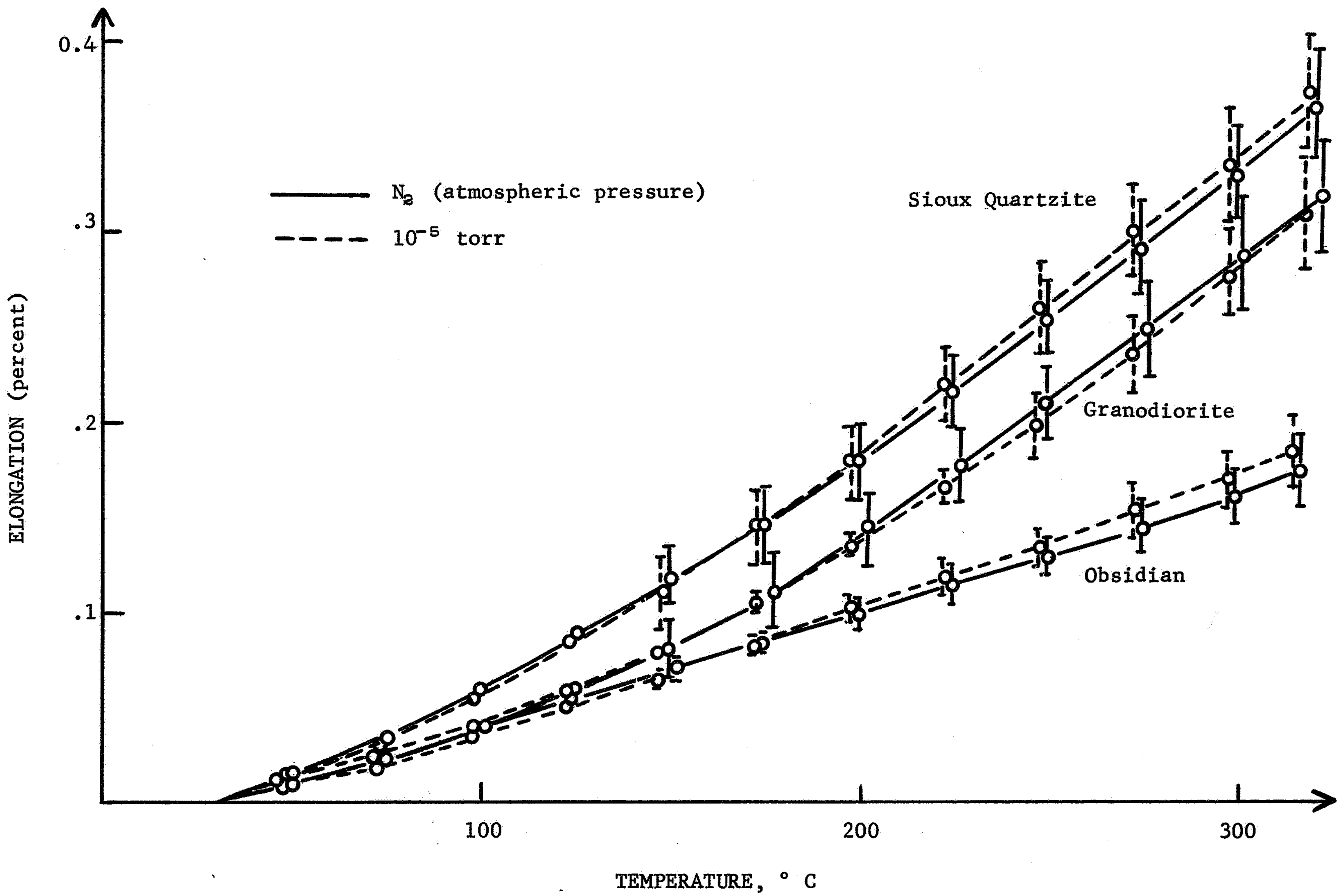
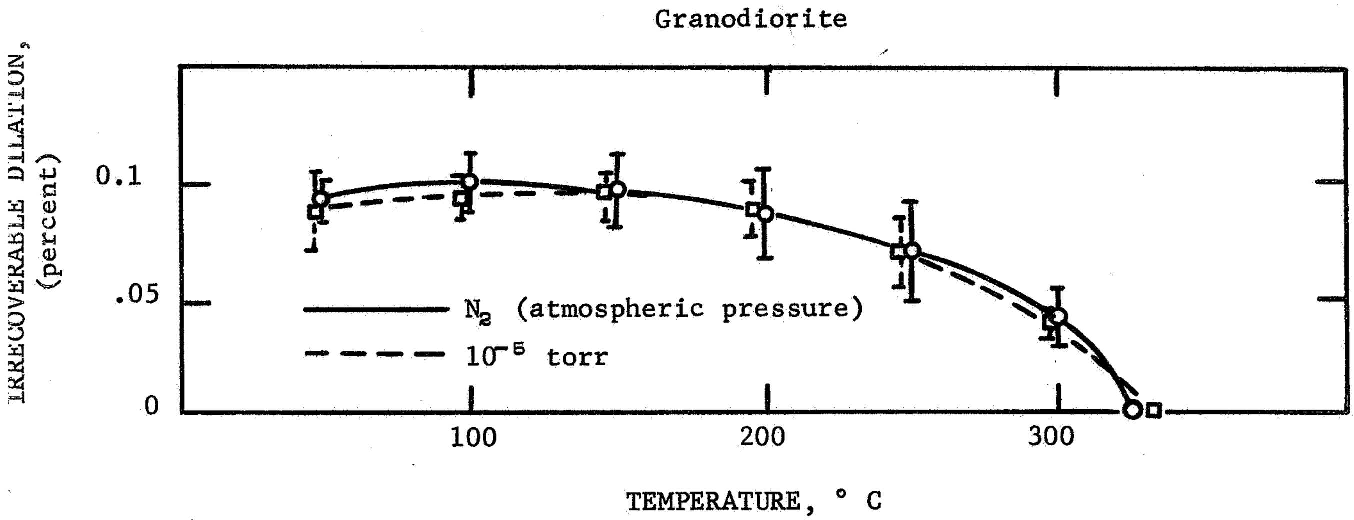


FIGURE 2. - Expansion Measurements.



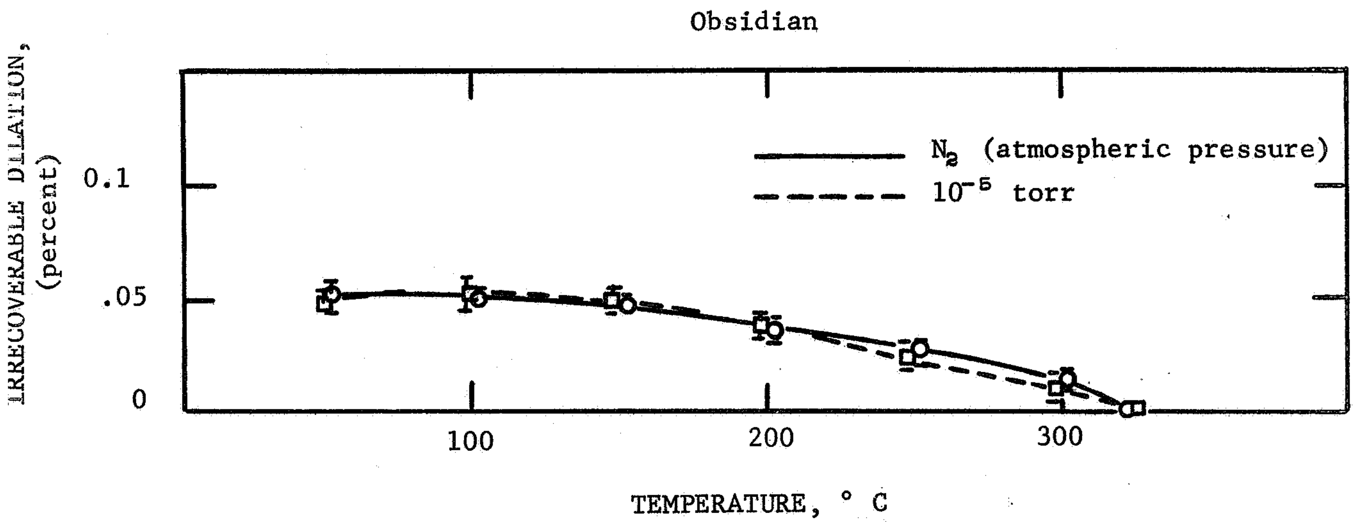


FIGURE 3. - Magnitude of Inelastic Dilation.

This study shows that the formation of internal thermal stresses during heating is largely dependent on the differences in thermoelastic properties of the rock material constituents. The internal thermal stresses create structural damage in the rock material. The thermal expansion and response of rock material to induced thermal stresses remain unaltered under reduced pressure down to 10^{-5} torr (10^{-3} N/m^2). These results were further confirmed by fragmenting thin test rock disks of each of the rocks with our CO_2 laser under both experimental conditions.

To examine the effect of the vacuum environment on a nonspalling rock, Dresser basalt was exposed to the laser beam under the two environmental conditions. In the nitrogen atmosphere melting occurred with little fragmentation. A self ejection of the melt was observed in the basalt under reduced pressure. Measurements of the depth of rock damage during melting with and without melt removal indicate that progressive removal of melt soon after its formation will increase rock disintegration by melting.

Status of Manuscripts

Effect of Reduced Pressure on Thermal Expansion Behavior of Rocks and Its Significance to Thermal Fragmentation by K. Thirumalai and S. G. Demou was presented at the 1970 Symposium on Thermal Expansion of Solids, Santa Fe, N. Mex., June 10-12.

Dielectric Constants and Dissipation Factors for Fourteen Rock Types Between 20 and 100 Megahertz by R. E. Griffin is under preparation as a journal article.

Thermal Expansion Measurements of Simulated Lunar Rocks by R. E. Griffin and S. G. Demou is under preparation as a journal article.

ANNUAL STATUS REPORT FISCAL YEAR 1970

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Use of explosives on the Moon
Investigator: Richard W. Watson, Project Coordinator, Explosives Research
Location: Safety Research Center
Pittsburgh, Pennsylvania
Date begun: July 1966 To be completed: June 1971

Personnel: Richard W. Watson, Research Physicist
Charles R. Summers, Research Physicist
John J. Mahoney, Laboratory Technician
Elva M. Guastini, Explosives Equipment Operator

PROGRESS REPORT

Objective

The objective is to develop fundamental knowledge relating to the hazards of the use of chemical explosives in the lunar environment, in particular, an environment characterized by high vacuum, extreme temperature cycling, and a flux of small hypervelocity particles.

Summary

The principal effort this year was devoted to the design and construction of a large-scale vacuum test facility for the purpose of extending our experimental charge weight capability which has been limited to about 50 g of high explosives. The new facility is designed to house a 38,000-liter cylindrical chamber and a 24,000-liter spherical vessel.

Progress During the Year

The construction of a large high vacuum facility suitable for firing up to several pounds of solid explosive at a vacuum of 10^{-4} torr or better has been completed. Two large chambers, a 3.6-m diameter (25,000 ℓ) sphere and a 2.1-m diameter, 11-m long (38,000 ℓ) cylindrical tank, have been installed, a 130-cfm mechanical roughing pump and a 20-inch oil diffusion pump have been procured, and a shelter erected around the system. Final design of the system and procurement of remaining components is in progress.

Fundamental knowledge of the behavior of detonation products in a simulated lunar environment has been advanced by means of time-resolved spectroscopy of the light emitted when the edge of the expanding products cloud impinges a solid surface in a vacuum. These experiments, using small expendable chambers, have verified that species separation does occur in the leading edge of the cloud and quantitative measurements have been made of the time of flight of readily identifiable species introduced by "seeding" the explosive. These measurements are expected to aid in the selection of alternative physical models of the process whereby the products are accelerated to velocities about double the expected value.

The expansion of a cloud of detonation products in a vacuum has been modeled theoretically and a computer program written for its solution. Models of the acceleration process are being incorporated into this program. The first of several acceleration processes to be considered is the ambipolar diffusion of electrons and positive ions which would cause single positive ions to acquire exactly double the velocity of corresponding neutral species. This is being tested experimentally.

Status of Manuscripts

None scheduled.

ANNUAL STATUS REPORT FISCAL YEAR 1970

Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Effect of lunar environment on behavior of fine particles
Investigator:	David E. Nicholson, Project Leader
Location:	Spokane Mining Research Laboratory Spokane, Washington
Date begun:	April 1966 To be completed: June 1971
Personnel:	David E. Nicholson, Mining Engineer William G. Pariseau, Civil Engineer Kelly C. Strebig, Mining Engineer (Twin Cities) Robert W. Carnes, Engineering Technician

PROGRESS REPORT

Objective

The objective is to determine the frictional and cohesive properties of simulated lunar rock powders which may influence the handling and transportation of fine particle materials in the lunar environment.

Summary

Programmed project work was completed except for two work items: (1) The torsional-shear tests on simulated lunar soil which were to be conducted in the Twin Cities ultrahigh vacuum chamber have not been performed as experiments with the simulated lunar drill have pre-empted the vacuum system; and, (2) planned low-stress, direct-shear tests on simulated lunar soil were deleted because of difficulties in adapting this form of shear testing to the low-stress range.

However, physical property testing of simulated lunar soil has been completed sufficiently so as to perform design analysis of gravity flow from bins and hoppers. Extensive review of research on bin flow (and of present methods for the design of gravity-flow bins) indicates that most of today's design-analysis methods have many deficiencies. Therefore, plans were made to adapt finite element analysis to the bin-flow problem.

Progress During the Year

The grinding and classification circuit completed in 1969 was used to produce simulated lunar soil by milling flow basalt rock from the Madras, Oreg. formation. The grain-size distribution of this simulated lunar rock powder parallels closely the grain-size distribution of the lunar soil samples obtained at the Apollo landing sites.

Torsional-shear tests under standard atmospheric conditions were completed during the year. To simulate lunar gravity conditions, this shear testing was done in very low-stress ranges. In addition, low shear-strain rates and large shear strains were possible with these tests. Analysis of the shear data and comparison of other physical property data indicate that the simulated lunar soil is a good descriptor, a close analogue, of actual lunar soil material. Completion of the shear test work will make possible the description of the yield surface of the simulated lunar soil. Previous work on consolidation and earth pressure will provide sufficient data to analyze the bin flow of this material.

Constructed and instrumented during the year were two models of the torsional-shear apparatus. One was assembled at the Twin Cities for use in the vacuum testing, whereas the other was built at Spokane for standard atmosphere testing of the simulated lunar soil.

The earth-atmosphere tests completed during the year consisted of thirty instrumented shear tests. The primary method was to shear the powder in several ranges of consolidation to determine any significant variation in its cohesion. Time-of-consolidation effects on the cohesion of the powder were minor, and increases in consolidation loads (up to 1 psi) also seemed to have little influence on the cohesion. The earth-atmosphere tests indicate an angle of friction of about 41°, and a cohesion intercept of about 11.5 g/cm^2, for simulated lunar soil consolidation loads up to about 90 g/cm^2. It will be important to observe whether these cohesion values are similar in the vacuum tests.

Status of Manuscripts

Testing of Simulated Lunar Basalt Powder for Gravity Flow by David E. Nicholson is under preparation as a journal article.

ANNUAL STATUS REPORT FISCAL YEAR 1970

Bureau of Mines NASA Program of Multidisciplinary Research

Task title: Electrowinning of oxygen from silicate rocks
Investigator: Donald G. Kesterke, Project Coordinator
Location: Reno Metallurgy Research Center
Reno, Nevada
Date begun: June 1966 To be completed: May 1970

Personnel: Donald G. Kesterke, Metallurgist
Delbert C. Fleck, Research Chemist
Freddy B. Holloway, Physical Science Technician

PROGRESS REPORT

Objective

The objective is to determine the feasibility of obtaining elemental oxygen by electrolysis of silicate-bearing materials, as one phase of multidisciplinary research efforts to develop basic knowledge for using lunar resources in support of space missions.

Summary

The experimental work on this project has been completed and a final report is being prepared. During the past 4 years, investigations were made of the operating characteristics of silicate-fluoride systems over a wide range of compositions to determine the relative electrical conductivity and the relative oxygen yield of the mixtures. Melts were evaluated that contained 25 weight-percent LiF, 40 to 75 weight-percent silicate rock, with the balance being BaF_2.

Results showed that the electrical resistance of mixtures containing more than 60 weight-percent silicate was too high for these to be considered workable electrolytes, and studies to determine the oxygen yield were limited to melts containing 40 to 60 weight-percent silicate. Experiments were performed at 1,100° to 1,200° C, at about 20 volts and 40 to 50 amperes, using iridium anodes and SiC cathodes. The maximum oxygen content was less than 40 percent of what was previously achieved using a bath composed of 35.0 weight-percent silicates, 16.5 weight-percent LiF, and 48.5 weight-percent BaF_2. This electrolyte has been shown to be superior to other systems studied, and was therefore used in all subsequent experiments.

Investigations were made to evaluate special cells for use in quantitatively determining the oxygen yield during electrolysis. The crucible was divided into an anode and cathode compartment by means of a perforated boron nitride barrier. A boron nitride lid over the anode compartment was designed to restrict the gases evolved during electrolysis to

a known volume, permitting calculation of the oxygen yield. In several experiments using this type of cell, it was shown that the presence of the barrier effectively eliminated excessive corrosion of the anode caused by contact between it and the cathode deposit. However, it was also found that the anode gases could not be completely restricted to the anode compartment, and that an unknown amount of these gases escaped. This prevented the quantitative determination of the oxygen yield by means of gas analysis, and efforts were initiated to make semiquantitative determinations of the oxygen yield by utilizing materials balance data combined with chemical analyses of the electrolyte and cathode products.

Progress During the Fourth Quarter

The objective of the work during the last quarter was to complete experiments designed to permit the semiquantitative determination of oxygen evolved during electrolysis of silicate-bearing mixtures, and bring to a conclusion the experimental phase of the research project.

Efforts were directed toward the preparation of oxygen from the mixture composed in weight-percent of 35.0 silicate, 16.5 LiF, and 48.5 BaF_2. Experiments were conducted that consisted of a series of 7 to 9 30-minute electrolysis periods. Electrolysis conditions were: a temperature of 1,100° to 1,160° C, a voltage of 17 to 19, and a current of 35 to 55 amperes. Iridium anodes and SiC cathodes were used, and after each period, the cathode and adhering deposit were removed and replaced by a new cathode. Accurate materials balance data were kept on the electrolyte and cathode products, and analyses of these materials were made from which the total decrease in oxygen content was calculated.

Data from the initial series of experiments showed that the oxygen content of the electrolyte before electrolysis was approximately 114 grams. During electrolysis a total of about 160 ampere-hours was applied to the cell, after which the combined oxygen content of the electrolyte and cathode products was approximately 97 grams. Assuming that the 17-gram difference in oxygen content was the result of electrolysis, this yields a calculated anode efficiency of almost 35 percent, based on an electrochemical equivalent of 0.29845 gram oxygen per ampere-hour.

Electrolysis experiments of extended duration were performed using the same experimental conditions and procedures as for the 30-minute periods. Electrolysis was conducted for a total of about 13 hours, during which time 567 ampere-hours were applied to the cell. Based on an electrochemical equivalent of 0.29845 gram oxygen per ampere-hour, the theoretical yield of oxygen was 169.2 grams.

Analytical data showed that the combined oxygen content of the baths and cathode products after electrolysis was 94 grams less than that of the electrolyte before electrolysis. This yielded a calculated anode efficiency of about 55 percent.

Anode losses during these experiments were somewhat greater than occurred earlier, possibly because scrap pieces of iridium were being reused as much as possible. A total of 45.4 grams of iridium was lost by electrochemical corrosion, or by reaction with the electrolyte or cathode products, for an average loss rate of 80 mg per ampere hour.

A report is being prepared describing the investigations carried out over the past 4 years and providing a basis for possible future work on electrowinning of oxygen from rocks.

Status of Manuscripts

Electrowinning of Oxygen From Silicate Rocks by Donald G. Kesterke was submitted to NASA for publication in the Proceedings of the Seventh Annual Meeting of the Working Group on Extraterrestrial Resources.

Electrowinning of Oxygen From Silicate Rocks by Donald G. Kesterke is being prepared as a Bureau of Mines Report of Investigations.